

## "TESTING METALS AND ALLOYS FOR USE IN OXYGEN SYSTEMS"

### INTRODUCTION

Oxygen is all around us. It is in the air we breathe. It is a part of nearly everything that we come into contact with in our daily lives. And even though it is critical to our very existence, there are hazards associated with oxygen that we are often unaware of. Oxygen is required for combustion. When oxygen is present in high concentration or high quantity, as in an oxygen system, then the likelihood of a fire starting and the intensity of the fire <sup>are</sup> increased. Many materials that are not considered to be flammable in air can ignite and burn vigorously in oxygen. For example, stainless steel, which is very difficult to burn in air, will burn vigorously when ignited in a 1000-psi oxygen environment [1]. Furthermore, a titanium alloy (Ti-6Al-4V), that is also very difficult to burn in air, will burn readily in an 8 psia 100% oxygen environment (See Figure 1) [2]. The hazards involved with the use of oxygen are increased when the system is operated at high temperatures. Ignition becomes more likely since the materials in the system are at elevated temperatures.

Advancing technology is creating a demand for higher oxygen-use temperatures and pressures. NASA is investigating the use of propulsion systems for a new generation of space-based orbital transfer vehicles (OTVs) that will use liquid hydrogen (LH<sub>2</sub>) and liquid oxygen (LO<sub>2</sub>) as propellants. A new design concept for the LH<sub>2</sub>/LO<sub>2</sub> engines involves the use of gaseous oxygen at 500°F and 5000 psi to drive the turbine in the LO<sub>2</sub> turbopump. Industrial gas suppliers are planning to increase the standard oxygen cylinder pressure from 2200 psi to 6000 psi. Higher oxygen-use temperatures and pressures such as these cause concern for engineers because they increase the risk of and damage caused by fire.

Fires in oxygen systems are generally catastrophic, causing damage to equipment and a threat to life. Such a fire occurred in April 1980, when a Space Shuttle extravehicular mobility unit (spacesuit and

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life-support system) was destroyed in a flash fire during a functional test in the Johnson Space Center (JSC) Crew Systems Laboratory (See Figure\* 2). A technician standing next to the unit received second-degree burns over his upper body in the accident. A board of investigation determined that the fire most likely started in an aluminum-bodied regulator in the oxygen system of the suit.

In another fire, a pressure-relief valve made of stainless steel was destroyed at the NASA JSC White Sands Test Facility (WSTF) in October 1973 (Figure 3). The fire, which started in the valve while it was operating at 9640 psi, destroyed the valve and damaged several other components in the oxygen system. In January 1961, an oxygen plant in Dortmund, Germany exploded, killing 15 persons and causing considerable damage to the plant and the surrounding buildings. The explosion was attributed to an oxygen leak. Because such fires are destructive and costly, those materials least likely to ignite and burn in oxygen must be determined so that safer systems can be designed. Engineers who design these new oxygen systems must carefully test and select the materials to be used in <sup>them</sup> ~~these~~ systems.

Although several test methods exist that can be used to determine the susceptibility to ignition of nonmetallic materials [3], until recently no methods existed that could be used to determine the susceptibility to ignition and burning of metals and alloys in oxygen. Test systems were needed that could be used both to obtain engineering data and to perform basic research concerning the ignition and burning characteristics of metals and alloys that could be used safely in oxygen systems.

At the NASA JSC White Sands Test Facility in New Mexico, LEMSCO is working to develop several test systems that can be used to evaluate metals and alloys for use in oxygen systems. Two of the test systems simulate ignition sources that have been known to cause fires in oxygen systems: frictional heating and particle impact. Two others simulate conditions that occur in oxygen systems once a fire has been started: flame propagation in static and flowing oxygen.

## FRICTIONAL HEATING TEST SYSTEM

When mechanical components of a piece of equipment rub together, heat is generated. If rubbing occurs in a system where oxygen is present in high quantities, even metals and alloys can be caused to ignite and burn. The heat generated by friction in ~~high-pressure~~ oxygen systems has been the cause of many fires. <

The frictional heating test system (Figure 4) has been developed to determine whether metals and alloys will ignite when their surfaces are rubbed together in oxygen. Two hollow, cylindrical test samples made of like or unlike materials are installed in the chamber. One of the samples is fixed to the test chamber and is stationary. The other sample is fixed to a rotating shaft. The shaft, which extends through the chamber, is connected on one end to a drive motor/transmission assembly capable of turning the rotary test sample at surface speeds ranging from 1 to 47 m/s (4 to 158 ft/s). The other end of the shaft is connected to an air cylinder that provides axial movement to the shaft and can be used to apply a normal force to the test samples of up to 4,400 <sup>N</sup> (1000 lb<sub>f</sub>). The chamber can be pressurized to 69 MPa (10,000 psi) with oxygen. The system is instrumented to measure the rotational speed of the samples, the load applied to the samples, sample wear, torque, temperature and chamber pressure, and emission of light from the samples. Ignition and subsequent burning of the test samples is determined by pressure and temperature changes, emission of light, and posttest examination of the test samples. <

To determine the susceptibility to ignition of a metal or alloy, the samples are installed in the chamber, the test pressure and the surface speed of the rotary sample are established, and the normal force is steadily increased until ignition occurs. The normal force per unit area ( $\text{N/m}^2$ ) at which the ignition occurs is multiplied by the surface speed (m/s) to give the power input per unit area ( $\text{W/m}^2$ ) required to ignite the material at the test conditions.

LEMSCO engineers and scientists have used the frictional heating test system to determine the relative susceptibility to ignition of several metals and alloys used in oxygen systems (Figure 5) [4]. The alloys toward the top of the list required a larger power input per unit area for ignition and were more difficult to ignite or had a greater resistance to ignition than those alloys shown at the bottom of the list. In general, the nickel-copper metals and alloys were shown to be more resistant to ignition than those alloys containing large amounts of iron and chromium. Also, metals and alloys that release smaller amounts of heat when they burn tended to be more resistant to ignition than those that release large amounts of heat.

When test samples made of different materials were rubbed together in oxygen, the pairs tended to ignite at the power input per unit area required to ignite the metal or alloy less resistant to ignition. That is, the metals and alloys that were easier to burn controlled the reaction, causing the metals and alloys that were harder to burn to ignite sooner than expected.

A series of tests was conducted to determine the effect of oxygen pressure on the power input per unit area required for ignition of Monel 400, stainless steel 316, and carbon steel 1015. In every case, as the oxygen pressure was increased above 1.7 MPa (250 psi), higher power input was required to ignite the samples (Figure 6) [5]. The fact that these alloys were more difficult to ignite at higher oxygen pressures runs counter to the popular view that higher oxygen pressures cause the likelihood of ignition to increase. While it is true that the amount of the material that was consumed in these tests was greater at higher oxygen pressures, the fact remains that they were more difficult to ignite at the higher pressures than at the lower pressures.

Does this indicate that when one is considering ignition by frictional heating that a system operating at high oxygen pressure is actually "safer" than a system operating at a lower oxygen pressure? This is the type of question that can be answered by data obtained from the frictional heating tester.

## PARTICLE IMPACT TEST SYSTEMS

What happens when small pieces of aluminum, entrained in a flowing stream of heated oxygen impact the seat of a flow control valve? In the fall of 1982, LEMSCO engineers at WSTF performed a series of tests to determine the answer to this question [6]. The pre- and posttest photographs in Figure 7 show the results of one of these tests. <sup>The valve was completely destroyed</sup> As a result of these tests, the valve was redesigned using alloys that were more resistant to ignition by particle impact than those that had been previously used in the valve. The new materials were chosen based on test results obtained using a preliminary version of the particle impact testers now being developed at WSTF.

Two testers are being developed to determine the resistance to ignition of metals and alloys that are subject to the impact of particles in heated oxygen flowing at various velocities. In the high-velocity or supersonic particle impact tester (Figure 8), a single particle is injected into a flowing stream of heated oxygen. The oxygen and the particle are accelerated to supersonic velocities as they pass through the converging and diverging nozzle. The particle impacts a target made of the test material as the oxygen flows around the target and is vented to the atmosphere.

In the low-velocity or subsonic particle impact tester (see Figure 9), up to 5 grams of particle material can be instantaneously injected into the flowing oxygen stream. The particle material is entrained in the oxygen and is carried through the test chamber where it impacts a target made from the metal or alloy being tested. The oxygen and particle material flow through holes on the periphery of the target and, finally, are vented to the atmosphere through the flow control orifice.

The WSTF high-flow gaseous oxygen test facility supplies heated oxygen to the test chambers at 7 to 38 MPa (1000 to 5500 psi) and ambient to 810 K (1000 °F) at flow rates up to 2.3 kg/s (5 lbm/s). Temperatures of the test gas and the target, and the pressure and the mass flow rate of

the test gas are measured. The velocity of the gas and the particles are calculated from this information. An ignition in both of the testers is detected visually and confirmed by posttest examination of the target.

By comparing the test sample temperature below which a material will not ignite, LEMSCO engineers have been able to rank metals and alloys for their susceptibility to ignition [7]. Figure 10 depicts the results of tests in which aluminum particles 1600 microns in diameter and traveling at supersonic velocity were impacted against targets made from 5 different alloys. The aluminum alloy was found to be the least resistant to ignition whereas Monel 400 was found to be the most resistant to ignition by particle impact. These results indicate clearly that the Monel alloy would be a much safer material to use in a gaseous oxygen system or component that could be subjected to particle impact.

The effect of test chamber inlet pressure on the temperature required for ignition of 316 stainless steel using aluminum particles 1600 microns in diameter is shown in Figure 11. As the inlet pressure was increased, there was a corresponding increase in the velocity of the particle, thus providing a greater kinetic energy to the particle. As a result, the target was ignited at a lower temperature as the pressure was increased. In fact, the particle had approximately the same kinetic energy, approximately 0.5 J, at the ignition threshold for each of the inlet test pressures used in these tests.

These results indicate that oxygen systems must be designed to limit the kinetic energy of particles that may be entrained in the flowing oxygen stream. Furthermore, the result can be used to establish temperatures, pressures, and flow rates at which systems made from various metals and alloys can be safely operated.

## FLAME PROPAGATION TEST SYSTEMS

Once a fire has started in an oxygen system, two questions come to mind: Will the fire spread or will it self-extinguish? And, if the fire spreads, at what rate will it do so? LEMSCO engineers at WSTF are developing two test systems designed to provide data that can be used to answer these and other important questions. In the first system, metals and alloys are tested inside a pressurized chamber in static oxygen. In the second, pipes or tubes made from various alloys are tested with oxygen flowing through them at various temperatures, pressures, and flow rates.

The static flame propagation test system can be used to determine the rate and extent of flame propagation of a metal or alloy in oxygen at pressures up to 69 MPa (10,000 psi). The test chamber (Figure 12) comprises a base and a top made from 316 stainless steel and has a volume of approximately 740 cm<sup>3</sup> (45 in<sup>3</sup>). The base has several electrical feedthroughs for igniter wires and thermocouples. The top has ports for connection to the oxygen supply and vent systems and three view ports through which the light from the burning test sample passes during a test. The chamber is protected from burning by a copper insert and baseplate. The test sample, a rod 0.3 cm (1/8 inch) in diameter is mounted vertically in the chamber with the igniter placed near the bottom or near the top of the sample, depending on whether an upward or downward propagation test is to be performed. The igniter, a thick-walled, hollow cylinder made of aluminum, is heated to its ignition point using a nichrome heating wire.

Prior to running a test, the test sample is cleaned and installed in the test chamber. The chamber is then purged and filled with 99.9% pure oxygen and the test pressure is established. Power is applied to the igniter heating wire. The igniter burns and the fire propagates to the test sample. As the fire propagates along the sample, the flame front passes by the view ports causing a large amount of light to fall on the thermopiles located outside each port. Since the view ports are 2.5 cm (1 inch) apart, these data can be used to calculate the flame propagation rate.

Engineers at WSTF have used this test system to determine the propagation rate of several metals and alloys in oxygen at various pressures [1]. Figure 13 gives results showing the effects of pressure on the flame propagation rates of 0.3 cm (1/8 inch) diameter samples made of six alloys. It was found that the propagation rate increased as the pressure was increased for each of the alloys that burned. Monel 400 burned only partially at pressures up to 55 MPa (8,000 psi) and copper 102 and nickel 200 did not ignite at the same test pressures. These data can be used by engineers to determine how quickly a fire may spread inside a pressurized oxygen system made from these alloys.

This system is also of particular interest to scientists who want to study combustion. A second chamber top has been made that has a view port 5 cm (2 inch) in diameter through which high speed photographs of the burning test sample can be taken. Obtaining a visual record of the burning event proves to be very helpful in understanding and predicting the behavior of burning metals and alloys. In conjunction with the University of California at Santa Barbara, WSTF scientists are developing an analytical model of flame propagation in metals and alloys [8]. The model, along with the data obtained from the test system, can in turn be used by engineers as a tool to aid them in designing safer oxygen systems.

The second of the two flame propagation test systems being developed can be used to determine the characteristics of flame propagation in a pipe or tube that is pressurized with flowing oxygen. This test simulates the conditions that occur when a fire burns through the wall of a pipe or a tube, venting the oxygen it contains to the atmosphere. Will the flame propagate along the pipe? If so, how fast will it propagate? What is the effect of the flow rate and pressure of the oxygen on the flame propagation? What is the effect of the thickness of the wall of the pipe or the configuration of the plumbing system on flame propagation? These questions and more can be answered using this test system. The system comprises an oxygen source - the WSTF gaseous oxygen high flow test facility - the pipe or tube being tested, an igniter, and high speed video and film equipment.



LEMSCO engineers have used this test system to determine the maximum flow rate of oxygen in 3/4 x .109 in. pipe against which a flame will propagate. Pipe made of 304 stainless steel was tested and it was found that at flow rates above 0.14 kg/s (0.3 lbm/s) the flame would not propagate upstream, against the flow of oxygen.

#### APPLICATION OF TEST RESULTS

Though still in the developmental stages, these test systems have already been used successfully in test programs for a variety of customers. As mentioned earlier, NASA used the particle impact test system in a study to determine if an oxygen flow control valve could withstand the impact of aluminum particles. The valve ignited and burned. As a result of further testing done at WSTF, new materials that were more resistant to ignition by particle impacts<sup>e</sup> were chosen for use in the valve. NASA's Lewis Research Center is sponsoring test programs at WSTF to obtain data to aid in the design of advanced concept propulsion systems for the OTV. Tests have been performed using the frictional heating and particle impact test systems to develop a data base on materials that may be used in those engines [9]. Frictional heating tests have been performed in liquid oxygen (LO<sub>2</sub>) to evaluate seal materials for use in LO<sub>2</sub> turbopumps in the OTV engines. <

The American Society of Testing and Materials in a joint effort with the Compressed Gas Association is sponsoring a test program to develop a data base for metals and alloys used in industrial oxygen systems. Frictional heating, particle impact, and flame propagation tests will be performed on several metals and alloys to determine their suitability for use in oxygen. NASA headquarters is making an effort to establish a criterion of acceptability for the use of metals and alloys in oxygen systems based on these test systems. The criterion of acceptability and descriptions of the required tests could be included in the agencies' document NHB 8060.1 [3], which establishes the terms of acceptability of a particular material prior to its use in manned space vehicles. agency)

Just as the test systems are being used to make important contributions to engineering, they will also be used to contribute significantly to scientific research regarding the combustion of metals and alloys. Because of their design, they can be used as tools for understanding the theories of the ignition and burning of metals and alloys. As a more complete theoretical understanding is obtained, advances in the state of the art of metals and alloys used in oxygen systems will be made. LEMSCO scientists are confident that the test systems will be used to provide data that will aid in the development of new alloys and surface treatments for metals and alloys. These improved, more burn-resistant metals and alloys will be used safely in the increasingly severe oxygen environments sure to be required in the future.

#### NEW TOOLS FOR ENGINEERS AND SCIENTISTS

Clearly, then, there is a great need for test systems that can be used to determine the suitability of metals and alloys for use in oxygen systems. LEMSCO engineers and scientists at NASA's White Sands Test Facility are developing test systems that meet this need. The test systems being developed have been used in test programs to provide data to engineers that enable them to design and build safer oxygen systems. Furthermore, they are being used by scientists to obtain a more complete understanding of the ignition and burning of metals and alloys. And finally, the test systems are available for use by both government and industrial users to provide data to answer questions and solve problems that will arise in the future regarding the safe use of metals and alloys in oxygen systems.

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### List of Titles For Figures

- Figure 1. Titanium alloy burns readily in 100% oxygen at 8 psia. Vertically mounted 1/8-inch diameter rod is ignited at the top and burns downward.
- Figure 2. This Space Shuttle extravehicular mobility unit was destroyed in a flash fire at NASA's Johnson Space Center in April of 1980.
- Figure 3. This stainless steel relief valve was operating under normal conditions in an oxygen system at 66 MPa (9600 psi) when it ignited.
- Figure 4. In the frictional heating test system, one test sample is mounted on the rotating shaft and the other is attached to the test chamber. A 15 H.P. drive motor and transmission assembly (see inset) powers the shaft and the normal load is applied to the test samples using a pneumatic cylinder.
- Figure 5. Power input per unit area required for ignition of metals and alloys at 6.9 MPa (1000 psi) in the frictional heating test.
- Figure 6. As oxygen pressure is increased above 1.7 MPa (250 psi) the power input required for ignition of Monel 400, stainless steel 316, and carbon steel 1015 is increased.
- Figure 7. Pretest and posttest views of a valve that burned as a result of particle impact. The valve was made using more burn-resistant materials as a result of these tests.
- Figure 8. In the supersonic particle impact tester a single particle is injected into flowing gaseous oxygen and is accelerated to high velocities as it passes through the orifice throat and diverging nozzle. The particle burns when it hits the target and, if the conditions are right, the target will ignite and burn.
- Figure 9. Many particles may be injected simultaneously using the subsonic particle impact testers. The particles are entrained in the flowing oxygen and are impacted against the target which is made from the material being tested.
- Figure 10. Copper and nickel bearing alloys are most resistant to ignition by particle impact while alloys that are high in iron content are more easily ignited and burned. Of the metals and alloys tested, aluminum was the least resistant to ignition.
- Figure 11. The ignition and burning of 316 stainless steel when impacted with an aluminum particle 1/16 inch in diameter was found to occur at a constant kinetic energy level.

### List of Titles For Figures (Continued)

Figure 12. In the static flame propagation test the sample is mounted vertically and ignited at the bottom. The flame front is detected as it passes the thermopile ports and the data from the thermopiles are used to calculate propagation rate.

Figure 13. As oxygen pressure is increased the flame propagation rate also increases.